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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the electronic ingredient with which a low dielectric constant, a low dielectric dissipation factor, and high toughness are required of both ends about the oligomer object of 2 functionality phenylene ether which has a phenolic hydroxyl group, and its intermediate field.

[0002]

[Description of the Prior Art] Toughness for a micro crack not to generate the mass data in a highly information-oriented society in the low dielectric characteristics for processing at high speed, a thermal shock, etc. is needed for the ingredient of the electrical and electric equipment and an electronic application. On the other hand, use of engineering plus CHIIKUSU, such as polyphenylene ether (PPE), is proposed. However, while PPE has the outstanding RF property, that compatibility with thermosetting resin, such as an epoxy resin and cyanate resin, is bad, melt viscosity's being high and fabrication nature's being bad, and the dissolving solvent being limited to halogenated hydrocarbon systems, such as aromatic hydrocarbon systems, such as toluene, benzene, and a xylene, or a methylene chloride, and chloroform, and having troubles, like workability is bad is known.

[0003] Although an approach, examination of false IPN structuring of PPE and cyanate resin, etc. (JP,11-21452,A etc.) which improve with the blend with other resin as a compatibilizer are made for the compatibility improvement, fabrication nature and thermal resistance are not solved. Moreover, for the moldability improvement, examination of the approach of making Macromolecule PPE low-molecular etc. is made. For example, the approaches (JP,9-291148,A etc.) to which a giant molecule PPE and a divalent phenol are made to redistribute under a radical catalyst, or the approach (JP,8-011747,B) of carrying out the oxidation polymerization of a divalent phenol and the univalent phenol is learned. A macromolecule object exists and neither can acquire 2 functionality low-molecular oligomer object efficiently.

[0004]

[Problem(s) to be Solved by the Invention] This invention is offering 2 functionality phenylene ether oligomer which has the electrical property and toughness which was excellent in PPE, is resin which has improved compatibility with other resin, and fabrication nature, dissolves in general-purpose ketone solvent in addition, and has the PPE structure qualification of an end phenolic hydroxyl group being

easy.

[0005]

[Means for Solving the Problem] As a result of repeating research wholeheartedly about 2 functionality phenylene ether, this invention person etc. discovers that it is carrying out the oxidation polymerization of the divalent phenol of a structure expression (2), and the univalent phenol of a structure expression (3) in ketones, and a structure expression (1) can manufacture efficiently 2 functionality phenylene ether shown with a structure expression (1), and came to complete this invention. Below, this invention is explained at a detail.

[0006] as shown in the following structure expression (2), R1 and R2 are the same as that of the divalent phenol of this invention -- or you may differ and they are a halogen atom, a with a carbon number of six or less alkyl group, or a phenyl group. R3 is a divalent phenol which has an upright same or biphenyl frame with indispensable you differing, it being a hydrogen atom, a halogen atom, a with a carbon number of six or less alkyl group, or a phenyl group, and R1 and R2 not being hydrogen atoms. [Formula 4]

[0007] In a structure expression (2), 2, 2', 3, 3', 5, 5'-hexamethyl-[1 and 1'-biphenyl]-4, and 4'-diol are desirable especially. When the divalent phenol which does not have a substituent in the 2nd place (R2, R3 of a structure expression (2)) is used for a raw material, since oxidizing velocity of a phenol own [divalent] is very high, a divalent phenol serves as diphenoquinone and deposits from a reaction solution. Priority is given to homopolymerization of the univalent phenol shown with the structure expression (3) as the result, and it progresses until the growth of the phenylene ether which has a phenolic hydroxyl group only at the piece end deposits from reaction mixture. Therefore, meltable 2 functionality phenylene ether is efficiently uncompoundable to a methyl ethyl ketone. For example, as a divalent phenol which does not have a substituent in the 2nd place, although 5, 5'-tetramethyl-[1 and 1'biphenyl]-4, and 4'-diol are mentioned, 3, 3', and when it compounds using this, the GPC spectrum of a sludge becomes like (drawing 1), and can check generation of a high molecular weight object. On the other hand, although 2, 2', 3, 3', 5, 5'-hexamethyl-[1 and 1'-biphenyl]-4, and 4'-diol are mentioned to the 2nd place (R2 of a structure expression (2)) as a divalent phenol which has a substituent From the GPC spectrum change under reaction at the time of using this divalent phenol (drawing 2), and transition (drawing 3) of average molecular weight, a reaction comes out of the molecular weight distribution of 2 functionality phenylene ether obtained from beginning to end, it is the same and generation of a high molecular weight object is hardly accepted. Therefore, the oligomer object of the target 2 functionality phenylene ether can be acquired efficiently.

[0008] Thus, when the divalent phenol which put the substituent into 2 and the 3 or 5th place was used, from the raw material which put the substituent into the 3 or 5th conventional place, the product of molecular weight distribution which did not attach anticipation, either was made. Therefore, in order to solve the technical problem of this invention, it is required to make the oxidation rate of a phenol own [divalent] relaxation, and it is indispensable to have a substituent in the 2nd place (R2 of a structure

expression (2)).

[0009] The univalent phenol of this invention is a univalent phenol shown with the structure expression (3).

$$\begin{array}{c|c}
R3 & R1 \\
\hline
 & -O-H \\
\hline
 & R3 & R1
\end{array}$$

in a structure expression (3), R1 is the same -- or you may differ and they are a halogen atom, a with a carbon number of six or less alkyl group, or a phenyl group. R3 is the same -- or you may differ and they are a hydrogen atom, a halogen atom, a with a carbon number of six or less alkyl group, or a phenyl group. It is desirable that what has a substituent especially in 2, the thing independence which has a substituent in the 6th place or this, the 3rd place, or the 3 or 5th place is used together. Furthermore, preferably, if independent, 2 and 6-dimethylphenol is good, and in concomitant use, 2 and 6dimethylphenol and a 2, 3, and 6-trimethyl phenol are good. As for the rate in the case of using together the univalent phenol which has a substituent in the 3rd place or the 3 or 5th place, less than [of the univalent whole phenol / 70mol%] is desirable. When [in the case of using together the univalent phenol which has a substituent in the 3rd place or the 3 or 5th place / than 70 mol% of the univalent whole phenol] more [comparatively], it becomes a crystalline compound and at least about 1000 mean molecular weight becomes insoluble at a methyl ethyl ketone. In addition, when a 2, 6-dimethylphenol and 2, 3, and 6-trimethyl phenol is mixed rather than the time of 2 and 6-dimethylphenol independent, a more low-molecular oligomer object is acquired. This is for the methyl group of the 3rd place of a 2, 3, and 6-trimethyl phenol controlling a polymerization, and suppressing generation of a high molecular weight object.

[0010] Next, the manufacturing method of this invention is explained. The oligomer object of 2 functionality phenylene ether shown with the structure expression (1) of this invention is acquired by carrying out the oxidation polymerization of the univalent phenol shown with the divalent phenol and divalent structure expression (3) shown with a structure expression (2). About the approach of oxidation, there is the approach of using direct oxygen gas or air. Moreover, there is also the approach of electrode oxidation. Which approach may be used and it is not limited especially. Since safety and plant-and-equipment investment are cheap, air oxidation is desirable. When oxidizing with air, as for a pressure, the pressure from atmospheric pressure to 20kg/cm2 is usually chosen.

[0011] As a catalyst in the case of carrying out an oxidation polymerization using oxygen gas or air Kinds, such as copper salt, such as CuCl, CuBr, Cu2SO4, and CuCl2, CuBr2, CuSO4, CuI, or two sorts or more are used. For the above-mentioned catalyst, in addition, Monod and dimethylamine, Monod, and diethylamine, Monod and a dipropyl amine, Monod and G n butylamine, Monod, and a -sec-dipropyl amine, Monod and dibenzylamine, Monod, and dicyclohexylamine, Monod and diethanolamine, ethyl monomethylamine, a methylpropyl amine, Allyl compound ethylamine, a methylcyclohexyl amine, a morpholine, methyl-n butylamine, Ethyl isopropylamine, benzyl monomethylamine, octyl benzylamine, Octyl-chloro benzylamine, a methyl (phenylethyl) amine, A kind or two sorts or more of amines are used together in benzyl ethylamine, a JI (chlorophenyl ethyl) amine, a 1-methylamino-4-pentene, a

pyridine, methylpyridine, 4-dimethylaminopyridine, a piperidine, etc. If it is copper salt and an amine, it will not be limited to especially these. Especially as an amine, G n butylamine is desirable. By using G n butylamine, homopolymerization of the univalent phenol shown with the structure expression (3) is controlled, and it is hard to generate a high molecular weight object, and becomes the oligomer object of 2 functionality phenylene ether with sharp molecular weight distribution.

[0012] Next, the solvent used for this invention is explained. In the oxidation polymerization, it is considered the poor solvent, and the ketone solvent and alcoholic solvent to which the rate which can be used in the oxidation polymerization of the conventional PPE was restricted can be used by this invention. Although neither the ketone of the reaction solvent to be used nor the rate of alcohol was able to be made [many] since this kind of reaction did not melt into an organic solvent but a **** polymer generated it conventionally, since the product of this invention was only low-molecular oligomer as shown in the above-mentioned chart (drawing 2), it was easily dissolved also in a ketone and alcohol, and the range of the solvent which can be used spread greatly. They can be used together with halogenated hydrocarbon solvents, such as aromatic hydrocarbons solvents, such as toluene which is the independence or conventional solvent, benzene, and a xylene, ethylene chloride, chloroform, and a carbon tetrachloride, etc. Although an acetone, a methyl ethyl ketone, a diethyl ketone, methyl butyl ketone, methyl isobutyl ketone, etc. are mentioned and a methanol, ethanol, a butanol, propanol, methyl propylene diethylene glycol, diethylene-glycol ethyl ether, butyl propylene glycol, propyl propylene glycol, etc. are mentioned as an alcohols solvent as ketones, it is not limited to these. When especially generation of the oligomer which is the purpose of this invention and which moreover shows a peak with sharp molecular weight distribution with low molecular weight comparatively uses a ketone solvent, the effectiveness shows up notably. The solubility of the divalent phenol which is furthermore a raw material to a use solvent has the most desirable partially aromatic solvent containing a methyl-ethylketone independent or a methyl ethyl ketone.

[0013] About the reaction temperature in the manufacturing method of this invention, although it is not especially limited if it does not go into the explosion limit of the solvent to be used, 25-50 degrees C is desirable. Since an oxidation polymerization is exothermic reaction, above 50 degrees C, temperature control is difficult and it is hard to carry out molecular weight control. Below 25 degrees C, it goes into the range of the explosion limit, and stable manufacture cannot be performed.

[0014] Next, the phenol concentration in the manufacturing method of this invention is explained. 2 - 20wt% of the concentration of the divalent phenol shown in the structure expression (2) is desirable to the dropped solvent. In the case of beyond 20wt%, a divalent phenol may not dissolve in a solvent completely. On the other hand, in the case of below 2wt%, the reaction rate of a polymerization falls. Moreover, 6 - 50 wt% of the concentration of the univalent phenol shown with the structure expression (3) is desirable to a solvent. When concentration is more than 50wt%, a univalent phenol may not dissolve in a solvent completely. On the other hand, in the case of below 6wt%, the reaction rate of a polymerization falls.

[0015] The mole fraction of the univalent phenol shown with the divalent phenol and divalent structure expression (3) shown with the structure expression (2) in the manufacturing method of this invention has the desirable range from 1:1 to 1:10. Especially, 1:2-1:8 are desirable. In this range, it is possible to be hard to produce homopolymerization of a univalent phenol and to perform molecular weight control. If the ratio of the univalent phenol shown with the divalent phenol and divalent structure expression (3) shown with the structure expression (2) is made fewer than 1:2, the residual of the divalent phenol

shown with the structure expression (2) will increase. Moreover, if a ratio is made [more] than 1:10, homopolymerization of the univalent phenol shown with the structure expression (3) will arise, and molecular weight will become large too much and will become insoluble oligomer at a methyl ethyl ketone.

[0016] The manufacturing installation and the manufacture approach of this invention are explained. A copper catalyst, an amine, and a solvent are taught to the longwise reactor which stirring equipment, a thermometer, air installation tubing, and a baffle plate attached, and it stirs at 40 degrees C, and the mixed solution of the divalent phenol beforehand dissolved in the solvent and a univalent phenol is dropped, performing bubbling of air. The range of drop time for 50 to 210 minutes is desirable. When there is no drop time in this range, distribution of the molecular weight distribution of the oligomer obtained becomes large. It is desirable to perform stirring from 5 minutes after dropping termination furthermore for 5 hours. Even if it performs stirring for 5 hours or more, since the increment in molecular weight does not take place, it should end the reaction further.

[0017]

[Example] Next, this invention is not limited by especially the following examples although this invention is concretely explained based on an example and the example of a comparison. in addition, measurement of number average molecular weight and weight average molecular weight -- gel permeation chromatography (GPC) -- it asked by law. Data processing was performed from the GPC curve and molecular-weight calibration curve of a sample. The molecular-weight calibration curve approximated the molecular weight of standard polystyrene, and the relation of elution time amount to the following formula, and obtained the molecular-weight calibration curve.

LogM = A0 X3+A1X2+A2 X+A3+A4 / X2 -- here, they are M:molecular weight, X:elution time amount -19, and A:multiplier. Moreover, the hydroxyl equivalent performed IR analysis (the liquid cel method) by having used 2 and 6-dimethylphenol as the standard substance, and asked for it from the absorption intensity of 3600cm-1.

[0018] To the longwise reactor of 2L which stirring equipment, a thermometer, air installation tubing, and a baffle plate attached, CuBr2 2.7g (0.012 mol), (Example 1) 70.7g (0.55 mol) of G n butylamine, methyl ethyl ketone 600g is taught. It stirs with the reaction temperature of 40 degrees C. divalent phenol "2, 2', 3, 3', 5, 5'-hexamethyl-[1 and 1'-biphenyl]-4, and 4'-diol" "I beforehand dissolved in the 600g methyl ethyl ketone -- " --g [55.7] (0.21 mols), 2, and 6-dimethylphenol 50.4g -- The mixed solution (mole fraction 1:2 of the univalent phenol shown with the divalent phenol and divalent structure expression (3) shown with a structure expression (2)) of (0.41 mol) is dropped over 120 minutes, performing bubbling of the air of 2 L/min. Furthermore, it stirred for 60 minutes after dropping termination, continuing bubbling of the air of 2 L/min. The ethylenediaminetetraacetic acid 2 hydrogen disodium water solution was added to this, and the reaction was suspended. Then, after the hydrochloricacid water solution of 1M performed washing 3 times, ion exchange water washed. The obtained solution was condensed by the evaporator, reduced pressure drying was performed further, and 100.3g was obtained. 650 and 810 hydroxyl equivalent weight average molecular weight were 310, and the number average molecular weight of this thing had them to the methyl ethyl ketone. [meltable] (This resin is described as "Ha" below.)

[0019] To the longwise reactor of 2L which stirring equipment, a thermometer, air installation tubing, and a baffle plate attached, CuCl 1.3g (0.013 mol), (Example 2) 79.5g (0.62 mol) of G n butylamine, methyl ethyl ketone 600g is taught. It stirs with the reaction temperature of 40 degrees C. divalent

phenol "2, 2', 3, 3', 5, 5'-hexamethyl-[1 and 1'-biphenyl]-4, and 4'-diol" "I beforehand dissolved in the 600g methyl ethyl ketone -- " --g [41.8] (0.16 mols), 2, and 6-dimethylphenol 75.6g -- The mixed solution (mole fraction 1:4 of the univalent phenol shown with the divalent phenol and divalent structure expression (3) shown with a structure expression (2)) of (0.62 mols) is dropped over 120 minutes, performing bubbling of the air of 2 L/min. Furthermore, it stirred for 30 minutes after dropping termination, continuing bubbling of the air of 2 L/min. The ethylenediaminetetraacetic acid 2 hydrogen disodium water solution was added to this, and the reaction was suspended. Then, after the hydrochloric-acid water solution of 1M performed washing 3 times, ion exchange water washed. The obtained solution was condensed by the evaporator, reduced pressure drying was performed further, and 111.4g was obtained. 1110 and 1450 hydroxyl equivalent weight average molecular weight were 580, and the number average molecular weight of this thing had them to the methyl ethyl ketone. [meltable] (This resin is described as "NI" below.)

[0020] To the longwise reactor of 2L which stirring equipment, a thermometer, air installation tubing, and a baffle plate attached, CuCl 1.1g (0.011 mol), (Example 3) 66.3g (0.51 mol) of G n butylamine, methyl ethyl ketone 500g is taught. It stirs with the reaction temperature of 40 degrees C. divalent phenol "2, 2', 3, 3', 5, 5'-hexamethyl-[1 and 1'-biphenyl]-4, and 4'-diol" "I beforehand dissolved in the 600g methyl ethyl ketone -- " --g [20.9] (0.077 mols), 2, and 6-dimethylphenol 75.6g -- The mixed solution (mole fraction 1:8 of the univalent phenol shown with the divalent phenol and divalent structure expression (3) shown with a structure expression (2)) of (0.62 mol) is dropped over 120 minutes, performing bubbling of the air of 2 L/min. Furthermore, it stirred for 30 minutes after dropping termination, continuing bubbling of the air of 2 L/min. The ethylenediaminetetraacetic acid 2 hydrogen disodium water solution was added to this, and the reaction was suspended. Then, after the hydrochloricacid water solution of 1M performed washing 3 times, ion exchange water washed. The obtained solution was condensed by the evaporator, reduced pressure drying was performed further, and 91.4g was obtained. 1700 and 2300 hydroxyl equivalent weight average molecular weight were 820, and the number average molecular weight of this thing had them to the methyl ethyl ketone. [meltable] (This resin is described as "HO" below.)

[0021] To the longwise reactor of 2L which stirring equipment, a thermometer, air installation tubing, and a baffle plate attached, CuCl 1.3g (0.013 mol), (Example 4) 79.5g (0.62 mol) of G n butylamine, methyl ethyl ketone 600g is taught. It stirs with the reaction temperature of 40 degrees C. divalent phenol "2, 2', 3, 3', 5, 5'-hexamethyl-[1 and 1'-biphenyl]-4, and 4'-diol" "I beforehand dissolved in the 600g methyl ethyl ketone -- "--g [41.8] (0.15 mols), 2, and 6-dimethylphenol 56.7g -- (0.46 mol) and a 2, 3, and 6-trimethyl phenol 21.1g (0.16 mols) mixed solution (mole fraction 1:4 of the univalent phenol shown with the divalent phenol and divalent structure expression (3) shown with a structure expression (2)), performing bubbling of the air of 2 L/min It was dropped over 120 minutes, and it stirred for 30 minutes after dropping termination further, continuing bubbling of the air of 2 L/min. The ethylenediaminetetraacetic acid 2 hydrogen disodium water solution was added to this, and the reaction was suspended. Then, after the hydrochloric-acid water solution of 1M performed washing 3 times, ion exchange water washed. The obtained solution was condensed by the evaporator, reduced pressure drying was performed further, and 111.9g was obtained. 1000 and 1350 hydroxyl equivalent weight average molecular weight were 520, and the number average molecular weight of this thing had them to the methyl ethyl ketone. [meltable] (This resin is described as "HE" below.) [0022] To the longwise reactor of 2L which stirring equipment, a thermometer, air installation tubing,

and a baffle plate attached, CuCl 1.3g (0.013 mol), (Example 1 of a comparison) 79.5g (0.62 mol) of G n butylamine, methyl ethyl ketone 600g is taught. It stirs with the reaction temperature of 40 degrees C. divalent phenol "3, 3', 5, 5'-tetramethyl-[1 and 1'-biphenyl]-4, and 4'-diol" "RO beforehand dissolved in the 520g methyl ethyl ketone -- " --g [37.4] (0.16 mols), 2, and 6-dimethylphenol 75.6g () [0.62] The mixed solution (mole fraction 1:4 of the univalent phenol shown with the divalent phenol and divalent structure expression (3) shown with a structure expression (2)) of mol is dropped over 120 minutes, performing bubbling of the air of 2 L/min. Furthermore, for 30 minutes after dropping termination, when stirred continuing bubbling of the air of 2 L/min, a lot of precipitate in a reaction solution was obtained. The ethylenediaminetetraacetic acid 2 hydrogen disodium water solution was added to this, the reaction was suspended, and the solid was filtered. Then, the methanol performed washing for the obtained solid 3 times. The obtained solution was condensed by the evaporator, reduced pressure drying was performed further, and 80.1g was obtained. 5300 and 9000 hydroxyl equivalent weight average molecular weight were 3800, and the number average molecular weight of this thing had them to the methyl ethyl ketone. [insoluble] This resin is described as "TO" below.

[0023] To the longwise reactor of 2L which stirring equipment, a thermometer, air installation tubing, and a baffle plate attached, CuCl 1.3g (0.013 mol), (Example 2 of a comparison) Pyridine 48.7g (0.62 mol), methyl ethyl ketone 600g is taught. It stirs with the reaction temperature of 40 degrees C. divalent phenol "2, 2', 3, 3', 5, 5'-hexamethyl-[1 and 1'-biphenyl]-4, and 4'-diol" "I beforehand dissolved in the 520g methyl ethyl ketone -- " --g [41.8] (0.16 mol), 2, and 6-dimethylphenol 75.6g -- The mixed solution (mole fraction 1:4 of the univalent phenol shown with the divalent phenol and divalent structure expression (3) shown with a structure expression (2)) of (0.62 mol) is dropped over 120 minutes, performing bubbling of the air of 2 L/min. Furthermore, it stirred for 30 minutes after dropping termination, continuing bubbling of the air of 2 L/min. The ethylenediaminetetraacetic acid 2 hydrogen disodium water solution was added to this, and the reaction was suspended. Then, after the hydrochloricacid water solution of 1M performed washing 3 times, ion exchange water washed. The obtained solution was condensed by the evaporator, reduced pressure drying was performed further, and 110.2g was obtained. 1100 and the 1820 hydroxyl equivalent weight average molecular weight of the number average molecular weight of this thing were 600. (This resin is described as "CHI" below.) [0024] The above result was summarized in Table 1. By making [many] the mole ratio of a divalent phenol from examples 1, 2, and 3, number average molecular weight and weight average molecular weight become large, and 2 organic-functions oligomer which has the molecular weight distribution of a wish can be obtained by changing a mole ratio. an example -- two -- a comparison -- an example -- one -- a result -- from -- divalent -- a phenol -- three -- place -- a substituent -- not having -- a biphenyl -- a phenol -- "-- three -- ' -- five -- ' - tetramethyl one - [-- one -- one -- ' - a biphenyl --] - four -- four -- ' - diol -- " -- a raw material -- using -- if -- with a mean molecular weights of 5000 or more oligomer -- generating -- 2 functionality phenylene ether meltable to a methyl ethyl ketone -- efficient -being uncompoundable.

[0025] That is, it is indispensable for compounding meltable 2 functionality phenylene ether efficiently to a methyl ethyl ketone to have a substituent in the 3rd place of a divalent phenol. If G n butylamine is used for an amine from the result of an example 2 and the example 2 of a comparison, the oligomer which has molecular weight distribution more sharp than the time of using a pyridine will be obtained. When a 2, 6-dimethylphenol and 2, 3, and 6-trimethyl phenol is mixed rather than the time of using 2 and 6-dimethylphenol independent for a univalent phenol from the result of an example 2 and an

example 4, more low-molecular oligomer is obtained. This is because the methyl group of the 3rd place of a 2, 3, and 6-trimethyl phenol controls a polymerization and generation of a macromolecule object is suppressed.

[0026]

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JP,2003-012796,A [DETAILED DESCRIPTION]



[0027]

[Effect of the Invention] The oligomer object of 2 functionality phenylene ether of this invention is fully meltable to the solvent of a ketone system, well, the varnish for laminates can adjust easily and compatibility with thermosetting resin can manufacture the charge of a laminated wood which is excellent in fabrication nature. Since it is one polyphenylene ether of engineer plus CHIIKUSU in which a basic frame unites and has low dielectric characteristics and toughness, it becomes a PPE polymer, and the electrical and electric equipment and electronic ingredient which have the same property. Furthermore, the reaction of degeneration of an end phenolic hydroxyl group can carry out easily in ketone solvent.

[Translation done.]